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Report No. G-1.133.3

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CLINTON ENGINEER WORKS - TENNESSEE EASTMAN CORPORATION
Medical Division

TITLE: PILOT SCALE COLLECTION AND RECOVERY OF AIR-BORNE TUBALLOY IN BLDG. 9206

ABSTRACT: There is included in this report methods for evaluating and collecting Tuballoy dust in the above building. In addition there is presented an analysis of the procedures to be followed in recovering Tuballoy dust from the type of collectors here proposed.

On the basis of work done to date the annual losses of T are estimated to be as follows:

Bldg.	9206	31.7 kg/year
	9204-1	13.9
	9204-2	48.3
	9204-3	7.2 (approximation)
		102.1

Further work on collectors of higher efficiency is under way, but the methods outlined in this report apply and will require no modification.

AUTHORS: Stanton B. Smith and William I. McPherson
9706-2

MS/CHL 2-0048/DEL REV
~ 50 Kg/yr. (365 Days/yr)
~ 36 " (264 Days/yr)

Date Issued: November 19, 1945

361.7

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PILOT SCALE COLLECTION AND RECOVERY OF AIR-BORNE TUBALLOY IN BLDG. 9206

PURPOSE AND SCOPE OF THE STUDY

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PRACTICAL DUST COLLECTION

GENERAL CONSIDERATIONS

An attempt will be made in this section to point out some design details worthy of consideration for the practical collection of dust.

The air volumes handled in the pilot plant runs were about 170 CFM, while about 800 to 1,000 CFM air should pass through a filter under normal operating conditions. The efficiency would decrease to some extent because of the greater velocities, but Mr. DallaValle believes it would be only slightly less efficient initially and the efficiency would increase rapidly with time as the deposit accumulated.

Early in this project of dust investigation it was realized that the dust concentrations originated at very definite dust sources and that the most efficient method of handling the problem would be the isolation of the sources and filtering of the exhaust air from the enclosed sources. In Room 6 the main dust sources are the carbon breaking operation and the removal of ash-laden trays from the muffle furnaces. If these operations were isolated and equipped with a suitable exhaust and filters, the Tuballoy dust concentrations in Room 6 would drop to a desirably low level and almost all of the Tuballoy dust would be caught on the filters. Thus, the relatively difficult problem of filtering all the exhaust air from the room would be resolved into the comparatively simple operation of filtering exhaust air from a small enclosed area. A total of 78,000 CFM air is exhausted from the rooms listed in Table V. At a maximum air volume of 1,000 CFM passing through one PL 24 filter, a total of 78 units would be required for those rooms. If channeling or isolation methods were used to aid dust collection at the source, a small fraction of the 78 units would be required to remove practically all of the collectable dust from 9206.

Another point worthy of consideration in the matter of collection is that dust is dispersed mostly in dry operations and not in most wet operations. The air from dry operations is not filled with acid fumes and, thus, the paper filter does not undergo chemical attack. However, if a paper filter were put in a hood exhaust (where mainly wet operations such as leaching and evaporation are carried out) the attack by fumes would rapidly result in the destruction of the paper filter. Then by enclosing the dry dust sources, the bulk of collectable dust would be caught on the filter without consideration of acid fume attack. Of course, glass wool filters could be used to collect dust in the presence of acid fumes. A study of the use of this material has not been carried out, but could be easily investigated with our equipment if it seems desirable.

A second major dust source that can be taken care of quite simply is settled dust. Settled dust is easily dispersed in the air both by air movements and by human activity. If the settled dust were cleaned up at frequent intervals and saved for processing, this second major dust source could be easily eliminated.

It is evident that by enclosing dustyoperations and removing settled dust the most important parts of the dust problem are solved.

At a recent meeting of several persons interested in dust collection it was generally agreed that electrostatic means of precipitation would require much expensive equipment that would be difficult to maintain. Also, the recovery of Tuballoy from oils used in the precipitation was suggested to be quite difficult and impractical for salvage. For these reasons pilot runs on this type of equipment were not carried out. However, along these lines it was suggested that an electrostatic ionizer combined with a grounded filter be used for dust collection. Such a unit is commercially available and should be on hand soon for pilot runs. This type of collector would be especially valuable in places where concentrations and particle sizes are low but where the Tuballoy is thought worthy of collection.

To be sure, after the adoption of the two suggestions for keeping dust concentrations to a minimum, there will be a small residual concentration of T in the air, but the collection of this Tuballoy probably would not be economically feasible.

SUMMARY OF RECOMMENDATIONS

1. Filter units of the PL-24 type loaded with 10 ply treated filter paper should be considered as a practical means for collecting the bulk of the airborne Tuballoy dust in Bldg. 9206.

2. Dusty operations should be carried out in a ventilated enclosure provided with suitable means for collecting the dust created.

3. All surfaces should be cleaned frequently with a vacuum cleaner and the collected dust should be processed.

4. The feasibility of processing air containing low concentrations of Tuballoy, and Tuballoy found in the smaller particles should be considered. The proper authorities should decide on how far the program should be carried in this direction.

It is hoped that the material presented in this report will be of help in considering the dust problems found in Y-12 and will offer some suggestion in regard to the practical collection of dusts and subsequent recovery of Tuballoy.

S. B. Ingram

W. H. McPherson

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PURPOSE AND SCOPE OF THE STUDY

This report summarizes and brings to date the record of investigation of Tuballoy dust concentrations conducted by the Industrial Hygiene Section of the Medical Division and Mr. J. M. DallaValle, Industrial Hygiene Engineering Consultant. The purpose of the study is first to determine the sources and magnitude of air contamination, and then, as indicated, both from the industrial hygiene and recovery viewpoints, consider and recommend methods for prevention, control, and recovery of the contaminants.

Initial studies were made in Buildings 9202, 9201-1, 9204-1, and 9206 and the results are summarized in the reports submitted by Mr. DallaValle on July 5, 1945 (Med-197) and August 21, 1945 (No. G-3.200.1). The primary function was the evaluation of the health hazard from Tuballoy dust, but it became apparent, from the amounts of Tuballoy involved and lost through ventilation, that the recovery for production purposes might be of considerable importance. After discussion of the preliminary results with Mr. Leo G. Warren and Dr. James G. McNally, it was agreed that the problem of recovery of Tuballoy from the air would be given equal emphasis in the study, and that control measures recommended would include suggestions on methods of collection and recovery of the air contaminants. In this study, as in most of the Beta areas investigated, the recovery for production has been emphasized, since the amounts of Tuballoy involved are in general below the levels hazardous to health, but of considerable economic importance.

Although the recovery of Tuballoy from the air is not the major function of the Industrial Hygiene Laboratory, at the request of Mr. Warren and Dr. McNally this activity will be continued. The inseparable character of the industrial hygiene and recovery problems, the special service and knowledge in evaluation and engineering control brought by Mr. DallaValle, the facilities for determining atmospheric contamination available through the Industrial Hygiene Laboratory, and the initiation of these studies by this group justifies this decision.

This report carries the investigation through two pilot plant scale recovery runs carried out in Room 6, Bldg. 9206, where our previous survey showed the highest concentration of beta recycle material. It was felt that engineering data could be obtained more rapidly in this location than in any other place in the refining division. It was also felt that the high concentrations of carbon, iron and other contaminants in salvage material would make a better test of recovery methods than would be obtained in other locations having dust of a relatively higher U content. In the two trial runs described, two types of special filter paper commercially available for industrial air filter installations were used.

Further tests are to be made using the same equipment in the beta building and possibly other locations in 9206. Additional equipment combining electrical precipitation and filtration methods is expected on the area soon. Data on its effectiveness will be obtained and compared with that contained in this report at a later date.

REVIEW OF ANALYTICAL METHODS

Previous reports have been concerned with the extent of losses in process buildings. An attempt is made in this report to show what proportion of material now found in the air may actually be reclaimed. In order to estimate these quantities with any degree of accuracy it is essential that reliable sampling techniques be used. At the beginning of the program most methods of estimating air concentration were open to question. However, we have always tried to use the best quantitative methods available at the time. Certain inconsistencies may be noted in comparing this report with figures previously reported; however, it should be kept in mind that consistency has been sacrificed through changes in method in order to report the best available data at the earliest possible date. A discussion of analytical techniques is given below.

DUST COLLECTION METHODS

Several methods of collection have been tried to date and several others carefully studied. Methods commonly used in this type of work are:

1. Electrostatic Precipitation
2. Filtration
3. Impingement

Electrostatic Precipitation

Measurements made in most of the early work in this program were made by this method. An MSA precipitron was used in conjunction with stainless steel tubes lined with tinfoil. This was the accepted type of collector as recommended by the Medical Research Section of the Manhattan District. This instrument uses a stainless steel tube about 1½" in diameter and 7½" long, in the center of which a wire electrode is suspended. A corona discharge is maintained on this electrode by means of a spark coil, condenser and rectifier. The voltage is kept at about 15 KV. Air is drawn into the tube at a rate of 3 cubic feet per minute, or a linear velocity of about 100 linear feet per minute. Dust and fume particles are deposited on the inside walls of the tube from the combined effect of the electric wind from this corona discharge and electrostatic attraction of ionized particles.

The electrostatic precipitator was at first thought to be almost 100% efficient for all types of dust over a wide range of particle size; however, it has since been shown that there are some dusts and fumes which are not collected well by this means. Lead, for instance, has been shown to be collected with an

efficiency as low as 70% at 15 KV at 480 LFM. Efficiency approaching 100% was reached only at 25 KV.[1] Other types of dust may be sufficiently charged before ionization so as to resist the electrostatic field and pass on through the collector. Some particles are also attracted to the center electrode where they cannot be "counted" easily. We have often noticed that in cases where dust concentrations are heavy that a deposit of material such as is found in Y-12 buildings will be found in the collecting head on the insulators and interior of the tubes beyond the dust collector tube. This indicates that material is being lost and it is likely that some material is also lost when concentrations are lower. Large particles may sometimes pass through the precipitron because of their greater inertia or they may sometimes be jarred loose from the tube wall even after precipitation and pass through. A relatively small number of such large particles will greatly outweigh a large number of small particles efficiently collected.

Dygert et al[2] in a series of experiments comparing various collection devices for Tuballoy dusts found that from electrostatic precipitators operated at 20 KV showed an efficiency of only 57% as compared with filtration with H-45 paper, the most efficient collector found, as 100%. However, actual efficiency of the precipitator is probably somewhat better as used in the plant, as all these tests were made in relatively high dust concentrations (2 mg to 20 mg/m^3) and it is known that efficiency of precipitation improves as the concentrations are lowered. Most of the concentrations first reported on Bldg. 9202 and 9206 in the above mentioned reports were made with the precipitron and were corrected on the basis of an arbitrary efficiency rating of 70%.

Another serious limitation of the MSA precipitron, entirely aside from its collection efficiency but playing just as important a part in final evaluation, is the flowmeter device. On this particular instrument the vane-type flow meter is easily corroded and clogged, so that the volume of air sampled is incorrectly measured. It is difficult to check the accuracy of this flow meter because of its sensitivity to pressure changes.

Filtration

Before the report by Mr. Dygert[2] had been received on the relative efficiency of various means of collection, information was obtained from the Chicago section on a new collection device especially developed by them for Tuballoy dust measurements. This device is known as the "Filter Queen" based on the name of the commercial vacuum cleaner from which it is made. It is designed for use with an asbestos base filter paper of rather heavy weight. A piece of this paper about 4½ x 8 inches is formed into a cylinder, cemented, and placed in a brass support tube called the "bird-cage". One end is closed and

[1] Keenan, R. G., and Fairhall, L. T., J. Ind. Hyg. and Tox., 26, #7, 241-249, 1944

[2] Dygert, H. P., Sanford, R., Oberg, H., Report K-1532, "Relative efficiencies of dust sampling devices as applied to the collection of U dusts"

the whole assembly placed in a chamber connected through a flowmeter to the inlet of the vacuum cleaner. With the large inner surface of the paper cylinder exposed to incoming dust, rates of flow up to 10 CFM can be obtained. The flowmeter is a rugged vane-type meter which is not influenced appreciably by pressure differences and not easily thrown out of adjustment. This device has been checked against an accurate "iron case" oxygen meter and found to be correct within reading errors. Even though the reading error is probably 10% at about 4 CFM and perhaps 5% at 10 CFM, it is felt to be much more reliable than the delicate flowmeter on the MSA device previously mentioned.

A specially designed paper known as H and V, Type #8912, was provided by the Chicago group. A. Pfanstiehl[3] in his instruction sheet for the Filter Queen makes the following statement in regard to the paper:

> "It is similar to the type of filter developed for gas mask use, employing an especially fine asbestos fiber as the collecting agency. The proportion of asbestos fibers (with diameters in the range of 1 micron) is several times more than that used in gas mask paper, since higher resistance could be tolerated. Its efficiency is such that the penetration of particles is less than that observable with the device used to check this type of paper for the Navy; that is, less than 0.005% penetration of a smoke with an average particle size of about 0.4 microns diameter. A large portion of the collected material remains quite near the surface."

Dygert[2], in the comparison tests mentioned above, employed several types of asbestos fiber base papers. He states:

> "The H-papers were made of asbestos especially designed to retain dusts of small particle size and to offer low resistance at high rates of air flow."

It is significant that by experiment involving the collection in TF_4 dust atmospheres (chemically analyzed at 2 and 20 mg per cubic meter) of a median particle size of 0.56 microns that the H-45 paper showed an average efficiency of 123% as compared with Whatman #41 as 100%. Determinations were made by weighing paper discs before and after collection of dust samples. Dygert summarizes the results of his filter paper efficiency tests as follows:

	Filter Paper	Comparative Efficiency
Asbestos Base Papers	H-45	123
	H-42	114
	H-51	111
	H-49	108
	Whatman #41	100
	Whatman #42	98
	OR-1661-A	80
	Whatman #50	55

[3] Pfanstiehl, A., "Instructions and precautions in the use of the 'Filter Queen' tester and associated apparatus" MUC-JAS-50, Medical Physics Laboratory files.

He states that the usefulness of the asbestos papers is limited by the difficulty of recovering dust from the papers for analysis. However, this is not a significant objection in the method of evaluation used for this report, as recovery of material from paper is not necessary. It appears from all considerations that the absolute efficiency of the HV 8912 paper may be 99% or better. A new type of paper[4] of the same type is now available but not yet in use here which is described as having an efficiency of 99.95% with particles averaging 0.4 microns diameter. It differs from HV 8912 paper only in that it is waterproofed and has fewer loose fibers.

Impingement

Though two types of impingers are at hand in this laboratory, this method was never seriously considered. It was felt that the wetting of particles of the more insoluble Tuballoy oxides would be difficult and subsequent chemical analysis would be more difficult owing to the smaller amounts of air that may be sampled by this method (0.1 CFM and 1 CFM, respectively, for the two types). It would also be necessary to remove the Tuballoy from the solutions which might be employed as wetting agents.

Some of these objections are borne out by Dygert's experiments using the "Greenburg-Smith Impinger" for sampling TO_2F_2, one of the more soluble T dusts. Using water as a collection medium, this apparatus showed an efficiency of about 70%, compared with the H-61 paper as 100%.

Tests on the "Midget Impinger" on TF_4, a relatively insoluble dust, using water as the collection medium showed widely varying results with an efficiency ranging from 42 to 74%.

Choice of the Filter Queen Sampler

The Filter Queen dust sampler seems to be the best all-round means now at hand for dust collection for the following reasons:

1. It shows the highest collection efficiency of all collectors for actual Tuballoy dusts so far investigated when used with an asbestos base paper.

2. The dust is evenly deposited over the paper surface and the Tuballoy can be easily estimated by alpha count methods without removing any material from the paper.

3. High sampling rates are attainable, which is highly desirable where dust concentrations are low.

[4] Pfanstiehl, A., Memo to, "Users of the Filter Queen" Aug. 2, 1946, Medical Physics Laboratory files

4. The apparatus may be depended upon to give nearly 100% efficiency over a wide range of particle sizes and concentrations. A number of very small particles may pass through, but these would account for an insignificant amount of material on a weight basis.

5. The instrument is rugged and maintains a uniform rate of flow over a long period of time.

6. The flowmeter is sturdy, easily checked, and easily recalibrated.

METHODS OF TUBALLOY ESTIMATION

The methods usually employed for the estimation of T are as follows:

1. Gravimetric analysis
2. Electro-chemical methods
3. Fluorescence analysis
4. Colorimetric analysis
5. Radiation measurements

Gravimetric Methods

In order that a method for the estimation be practical with existing sampling procedures, it must be capable of estimating quantities of the order of a microgram to a milligram of T. This rules out the use of gravimetric or volumetric methods as a practical means of measurement. In the very beginning of the project an attempt was made to use microchemical methods, but it was found to be too cumbersome and inaccurate for practical use.

Electrochemical Methods

Of the electro-chemical methods, the polarographic method was investigated. At first, this appeared to have much promise, but was time consuming and required apparatus difficult to keep standardized and required the removal of the material from the collector. This ruled out its use with anything but the precipitron, impinger or cellulose base filters, which are not efficient collectors. Also, the purification of individual samples presented quite a problem, as copper and iron interfere unless special complexing agents are employed.

Electrometic titrations are ruled out for the same reason as other volumetric methods. Necessary preliminary purification would also be difficult.

Colorimetric Methods

Colorimetric analysis would require a relatively large amount of material. Its use would also be limited because of high concentrations of contaminants and the necessity of removing the material quantitatively from the collector.

Fluorescence Methods

The fluorescence method has been in use in this laboratory for some time in connection with other work and has been found to be very sensitive. However,

it also has certain limitations. With the aid of a photoelectric instrument built specifically for the purpose by Mr. Hugh G. Neil of the Medical Physics Laboratory, samples may be run rapidly and readings obtained over a range of several orders of magnitude, being capable of detecting .001 mg of Tuballoy. However, in the workable range the accuracy obtained is approximately 20%. Preliminary purification is not necessary if the dilution technique can be employed as recommended by Dr. Price et al[5] in his report of fluorescence analysis, but it is still necessary to remove the dust sample from the collector. Though the fluorescence method may not be applicable to atmospheric dust concentration measurements, it is very useful in evaluating settled dust as collected on greased petri dishes. All analyses of settled dust given in the first two reports on alpha buildings and 9206 were done by this method.

Radiation Methods

Methods involving the measurement of alpha radiation of the Tuballoy contained in the dust seems the most practical from all considerations. In the first place, the method is specific for Tuballoy, since other alpha emitters are not commonly employed on the area. The method also fulfills the sensitivity requirements.

The first attempts at alpha counting dust samples early in the project were not promising as the material was removed from the collector, brought into solution, electroplated onto a platinum plate and the rate of alpha emission measured. This method was not practical, as a quantitative method of plating had not been devised.

The Rochester section, however, came out with the idea of estimating the Tuballoy without removing the material from the collection tube. Their method employed stainless steel precipitation tubes lined with tin foil which could be placed directly in an ionization chamber and the discharge rate of a charged electrode placed in the center of the tube measured with a Dale vane-type integrating electrometer. The use of the precipitron with this method of estimation comprised the approved method for dust concentration measurement as set forth by the Medical Section of the Manhattan District in Rochester.

This method is satisfactory if carefully standardized against deposits of known amounts of the same material that is to be collected. This is difficult to do in practice. Since the total ionization is measured and since this is dependent on the length of path of the alpha particles, it is readily seen that agglomerates of particles, thick uneven deposits, and foreign material will cut the path length of the alpha particles and give low results.

An alpha particle counter device has several advantages because it is capable of recording each alpha particle as a separate entity. Coincidence of counts, of course, will cause low results but is negligible for small quantities of

[5] Price, Perretti & Schwarts, "Microfluorometric Determination of Tuballoy" CC-3985

material. Depth of deposit, self-absorption and foreign material will also affect the counter, but to a lesser degree than the ionization chamber. A geometry factor for the cylindrical chamber counter is obtained by counting standard samples.

This method will give an accurate answer over a wide range, providing a count of at least twice "background" (counting rate of blank tube due to noise and loose particles and fibers in the chamber) is recorded.

Such a counting chamber was designed and built by Mr. A. H. Dahl's Medical-Physics group to accomodate precipitron tubes. It gave very satisfactory results. When the Filter Queen was put into use a similar chamber was furnished by the Metallurgical Laboratory to accomodate the paper filter tubes. This chamber, known as the "Long Tom" may be used for both the metal precipitator tubes and the paper tubes. The device was used by the Medical-Physics laboratory in estimating all the dust concentration determinations in this study.

The penetration of fine particles into the matrix of the filter paper is an additional factor affecting the accuracy of the count, as the paper itself may cause some absorption of the alpha particles. However, it is felt that the bulk of material remains close to the surface and a factor has been determined to compensate for this effect. [3, 4]

Some difficulty was experienced in the early part of this work because of doubt as to the specific alpha activity of the different series materials in use in the plant. Only an approximation could be made where material of two different series were used in the same general location. Where doubt arose, the best logical approximation was made based on the operation, amount of material, and locations involved. However, the data contained in this report is calculated on the basis of 1300 recycle material which is now in process exclusively in the areas investigated.

Evaluation of Errors.

Using the foregoing data as a basis for selection of a suitable method for evaluating dust concentrations, the Filter Queen filter collector in conjunction with the cylindrical chamber alpha counter was chosen as the most reliable. This method is still short of perfection, but is considered satisfactory when the transient nature of the dust concentrations we are trying to measure is considered. A sample calculation is given below together with an estimate of the variability of each particular quantity.

$$c = \frac{r}{v \; k_1 \; k_2 \; K}$$

where:

c = Concentration of Tuballoy in micrograms per cubic meter

r = Counting rate registered on scaler minus the rate of background (determined daily on blank tube) in counts per minute
(0 to -2% error)

v = Volume of air sampled in cubic meters (± 10% error)

k_1 = Geometry factor for chamber determined with standard samples (.40, 2 to 5% error)

k_2 = Absorption factor to compensate for counts lost through imbedding of particles in the paper (.70, -5% to +10% error)

K = Specific alpha activity of the material being used in counts per minute per microgram for full solid angle (± 1% error)

Thus the maximum error of a single determination is found to be about 25%. However, it is likely in most instances that accuracy is much better than this, as good checks are obtained on duplicate samples. It is impossible to make a closer evaluation of the method without a time consuming study using dust chambers maintained at known concentrations. About as good a test as may be devised for evaluation the sampling methods is that contained in this report; the gravimetric reclamation on a rather large scale checked against estimates made from sampling data. Results of the second run, where careful estimates were made and salvage methods are more or less standardized, showed a recovery of 82.6%, which is remarkable considering the number of variables involved.

AIR VELOCITY MEASUREMENTS

All figures given herein and in previous reports on air volumes leaving specific areas are based on actual measurement. The instrument used for this purpose was the Alnor "Velometer" which is equipped with a variety of jets permitting its use for any type of air velocity measurement in a range of 50 to 3,000 LFM. The Velometer is calibrated to an accuracy of 3% of full scale deflection, which is sufficient for our purposes without further correction. Several velocity readings were taken on each room opening, the velocities averaged and multiplied by the cross-section of the opening. The sum of the resulting air volumes (CFM) for a given room have been reported and have been multiplied by the average dust concentrations found in the room to give the total T loss for the room.

Because of recent changes in operation, climatic conditions, and alterations of equipment, the measurement of air movement is subject to large errors. However, we have purposely strived to be conservative in our estimates of T loss. When one considers the localization of many exhaust ducts, one might expect to find losses considerably larger than those reported. We feel, however, that the figures presented indicate correctly the order of magnitude of T dust losses based on measurements in the field, which is all we have intended to do.

EXPERIMENTAL PART

GENERAL INFORMATION ON DUST COLLECTION RUNS

Equipment Used

In order that data obtained in this study might be applicable to the engineering of a plant scale installation, equipment was used capable of handling a single unit cell of a standard air filter. The filter was the "PL 24" type manufactured by the American Air Filter Corporation. Installations of this unit are used to filter incoming air in most Y-12 production buildings. It consisted of a heavy metal frame 24" x 24" into which fit two paper holder sections having interlocking fingers at opposite sides and support bars running between the ends of these fingers. A piece of special filter paper about 16 feet long and 2 feet wide was pressed between the two holders in such a way that the edge of the paper is held air tight all around between the interlocking fingers and a well supported pleated paper surface is presented to the incoming air. A layer of protective screen was removed from the inlet side of the commercial filter so that all air-borne material would impinge directly on the paper surface.

The filter section was mounted on a "Roto-Clone" self-contained unit especially adapted in the Process Improvement shops by Mr. Withers and Mr. Droher of Mr. Graham's group. It was altered in such a way that the air entered directly into the paper filter, then passed through the blower and out through a section of 5" flexible tubing into a straight section of 5" pipe about 10 feet long. All surfaces of the filter cell and the interior of the Roto-Clone were painted white so that any adhering material might easily be noted and removed at the end of a run. The blower was belt-driven by a 440 V, 3 phase ½ HP motor. In operation the unit was smooth and quiet and capable of maintaining a flow of 125 CFM through a 10 ply paper filter. The only difficulty experienced in over a month of continuous operation was a loosening of the drive belt, which perhaps caused a slight reduction in air flow.

Inferences about the power requirements of a large installation could not be made on any valid basis from this unit, as a higher resistance to air flow was encountered than would be found in a large installation because of the tortuous air path and small pipe diameters of this unit.

Location of Equipment

A satisfactory location for the experimental studies was found in Room 6, Building 9206. This room has been found previously to give consistent and relatively high concentrations of Tuballoy dust, and for these reasons was selected for the experiment. The operations in this room are those of

carbon breaking and burning, acid leaching, evaporation, and muffling of residues. Important sources of dust production are the breaking of carbons and the handling of trays of muffled ash. It is well to note here that if certain rather simple precautions were taken, the Tuballoy dust concentrations could be cut to a low figure.

Because of the crowded conditions in Room 6, the Roto-Clone dust collection unit was installed on the balcony over the carbon burners. This position was ideal from the standpoint of being away from possible tampering. However, some of the heavier particles in the air might settle before they reached this height, so that only the smaller particles would be subject to collection. This point should be kept in mind when considering the data obtained and when considering any dust collection system.

The air passing through the filter was exhausted back into the room for the purpose of making dust concentration measurements and velocity measurements at the exhaust outlet and also to simplify installation. The air volumes exhausted by the collector were a negligible portion of the total for the room, and the outlet was ten feet from the inlet so that the discharge of the unit introduced no significant dilution of the dust concentration in the room.

The data presented in this report was obtained during two experimental runs in the periods from September 13 to September 28 and September 29 to October 15. In the latter run a two day shut-down period was caused by power difficulties. A six-ply untreated cellulose wadding type filter (Untreated "Airmat") was employed in the first run, and a similar ten-ply oil impregnated paper (Treated 10-ply "Airmat") was used in the second run.

Tuballoy Dust Concentrations

During the pilot runs frequent determinations of Tuballoy dust concentrations were made. The concentrations at both the inlet to the Roto-Clone and the outlet were determined and are found in Table I. The average Tuballoy concentration during the first run was found to be almost double that found during the second run. A possible reason for this reduction in concentration was the installation of protective covers over the carbon breaking barrels, thus reducing the Carbon-Tuballoy dust concentration during the period of the second run.

Much significance should be attributed to the Tuballoy concentrations at the filter exhaust. It is evident that although the Tuballoy concentrations at the filter inlet vary widely, the concentrations at the exhaust remain remarkably constant, and variations lie within the experimental error of counting at these low values. Thus, with dust of the type found in Room 6 about 7 mcg/m^3 Tuballoy dust will pass through a six ply untreated filter and about 2.7 mcg/m^3 will pass through the ten ply treated paper. These test runs lasted only two weeks, so it would not be unreasonable to assume that if the filter were left in use for a longer period of time, the concentration at the outlet would drop. A filter of the type used in these experiments can successfully collect air-borne Tuballoy dust and reduce the concentration to a desirably low figure.

TABLE I

TUBALLOY CONCENTRATIONS AND FILTER EFFICIENCIES

Date	Conc. of T at filter inlet, mcg/m^3	Conc. of T at filter outlet, mcg/m^3	Efficiency %
RUN I			
9-26	34	7.1	79
9-28	42	7.2	83
Average Run I	38	7.2	81
RUN II			
10-2	29.6	4	86.6
10-4	16	3.2	79.9
10-6	45	4	91.0
10-8	6	0	100
10-11	8.1	1.6	79.8
10-15	18	3.4	80.8
Average Run II	20.5	2.7	86.4

Efficiency of Collector

The efficiency of the filters is, of course, an important factor and is found also in Table I. The efficiency is dependent on the Tuballoy air concentration because of the relatively constant exhaust concentrations. Nevertheless, it is worthwhile to note that the average efficiency for the untreated six-ply paper was 81% and that for the ten-ply treated paper was 86.4%. Also the efficiency had a tendency to increase during the experimental runs, so that higher efficiencies could be expected on longer runs.

Flow Rates in the Dust Collection Equipment

The velocity of exhaust gas was measured periodically in the 5 inch outlet pipe. In the first run a general average of 1,300 LFM was found and during the second run a more frequent measurement was made, as seen in Table II.

The decrease in flow rate seen in Table II may have been the result of increasing resistance offered by the accumulating dust layer. It also may have been due to a gradual loosening of the belt on the motor. In any case the flow rates were not cut down enough in a period of two weeks to give much serious difficulty in the collection of dust by this means.

The average air velocity at the inlet in the first ~~run was~~ 41 LFM and 46.5 LFM in the second run. An average volume of 165 CFM was handled by the machine in the first run and a volume of ~~185 CFM~~ was handled in the second run. Thus a significant portion of the air in the room was sampled so that these experiments are valuable for practical considerations.

Calculation of Tuballoy to be Collected

With the data collected here it was possible to make a calculation of how much Tuballoy should be picked up by the filters. If the material found on subsequent recovery were in the range of that based on these calculations, much confidence could be placed in the use of the Filter Queen sample along with ventilation data as a means of accurately estimating quantities of Tuballoy lost through air-borne dust. Also, such positive evidence would be of great value from the medical and health aspects.

Accordingly, calculations were made including concentrations, air volumes handled, and efficiency of the filter, giving a theoretical yield of 3.1 gms. for the first run and 1.89 gms. for the second run.

RECOVERY OF TUBALLOY FROM PAPER FILTERS

At a recent conference held on the matter of dust collection, the subject of recovery of Tuballoy from the dust collector was debated. Several present thought the matter of recovery to be so difficult that the recovery would not be feasible even though materials such as oily rags, filter papers, bits of wool, and rubber gloves were being handled regularly by the Salvage Department. An objection also raised was that the ash content of the filter paper would be prohibitive to the recovery of T. It will be shown in this section that simple methods of recovery of Tuballoy are suitable and that actual recovery was carried out without much difficulty.

Burning of Filter

The simplest method of getting rid of cellulose, by far the major constituent of the dusty filter, is to burn it. The paper filter is extremely cheap and is easily disposable. It also should be noted that the paper will burn readily; no expensive muffle furnace is necessary for the burning of this paper. A simple, inexpensive incinerator could be readily fabricated that would answer the cellulose removal problem that was suggested to be so difficult. In the pilot experiment the filters were burned in a makeshift oven made by inverting two porcelain developing trays. A small opening was provided to give a controlled supply of air to the fire. Destructive distillation products of the paper deposited on the pans, so that smoke containing Tuballoy was caught on the tarry distillation components. The tar was quite easily dissolved by methyl alcohol in cleaning and the alcohol was readily boiled off leaving a residue for ashing. A concentrated filter paper ash resulted that was much more suitable for muffling than the original paper. This later muffling should be carried out to remove all organic material and to convert all the T to T_3O_8.

Ash Content of Filter Papers

A complete investigation of the filter paper ash of untreated and treated filters was made in order to answer the question raised as to the feasibility of recovery on this account. The ash content of the untreated paper was 0.27% and the content of the treated paper was 0.37%. This ash amounted to only 5.9% of the total ashed dust weight in our two week runs and, of course, would amount to less in a longer plant operational run. On this account the ash content of the paper is a negligible factor. Also, we were very fortunate in having Mr. Max Hill of Dept. 195 give us a quantitative spectrographic analysis of the filter ash as well as the dust ash. The ash analysis of the treated filter paper appears in Table III.

None of the elements listed in Table III in the proportions given offer any difficulty in the carrying out of the salvage operation. Silica might offer

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TABLE II

AIR VELOCITIES AT DUST COLLECTOR EXHAUST

Date	Air Velocity of Outlet, LFM
9-29	1,600
10-2	1,500
10-4	1,700
10-6	1,600
10-8	1,625
10-11	1,500
10-13	1,450
Average	1,596 LFM

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TABLE III

SPECTROGRAPHIC ANALYSIS OF FILTER ASH

Constituent	Analysis, %
Ca	50.
Si	2.
Al	5.
Fe	2.8
B	.03
Mn	.04
Mg	.8
Pb	1.0
Ni	.04
Cu	.2
Cd	.01
Zn	.2
Sn	.05
Cr	.1

some difficulty if it were a major constituent, but at a low level it is of not much significance. The paper ash can be readily put into nitric acid solution for the purpose of subsequent extraction of Tuballoy from this gunk. It is evident that no difficulty is presented to a salvage operation by the filter paper ash.

Analysis of Dust

A complete analysis of the dust collected on the filter in the 2nd run was made so that some idea could be gained about the ease and most suitable methods of Tuballoy recovery. The ash of dust picked up by the filter was 6.25% of the weight of the filter. However, 9.1% of the total ash was dust ash and the remaining 6% was filter ash. The spectrographic analysis of this combined ash is found in Table IV.

Many valuable and pertinent conclusions can be drawn from the spectrographic data in Table IV. First, the individual analyses, considering the elements to be present as the stable oxides, add up to about 96%. This means that the analysis is a very accurate one when considering the errors involved in the spectrographic method. This accurate analysis along with the proper interpretation of the analytical data offers a firm basis for setting up a salvage procedure.

The analysis shows that the biggest dust components are the constituents of concrete and ordinary dust. Also the components of stainless steel form an important fraction of the dust, with welding fume components present to a lesser extent. The carbon in the dust, of course, was lost when it was muffled and so does not appear in the analysis. Carbon is removed in the first salvage step of muffling and is of no importance in considering treatment of the ash for Tuballoy recovery. Tuballoy is present in the ash in sufficient quantity to make salvage worthwhile.

The nitric acid leaching method for putting the Tuballoy present in the ash into solution is suitable except for the presence of silica. This difficulty, of course, is encountered in the salvage department at the present time in many of the residues they are handling. Two methods are suitable for removing U from acid-insoluble silica residues. The first method is fusion with a suitable flux. The second is a removal of the undesired silica by treatment with hydrofluoric acid. Both of these methods work. The method used at the present time in salvage is that of repeated leaching with nitric acid. This method is neither as clean-cut nor as complete as the fusion or hydrofluoric acid treatments. These methods could prove to be simple and practical if put into operation properly.

Method of Recovery of Tuballoy from Dust

The method used with success for recovering Tuballoy from the air filters used in the two experimental runs is described below. The filter paper was removed from the holder and placed in a container made by inverting two porcelain trays. The paper was ignited and burned to the ash. This ash was

TABLE IV

SPECTROGRAPHIC ANALYSIS OF DUST ASH

Substance	Percentage of Component as the Stable Oxide	Percentage of Component as the Element
Ca	28.	20.
Si	30.	15.
Al	9.5	5.
Fe	14.3	10.
B	.02	.006
Mn	.03	.02
Mg	.40	.24
Pb	.12	.1
Ni	.26	.2
Cu	2.6	2.0
Cd	.05	.04
Zn	2.4	2.
Sn	1.3	1.
Cr	.3	.2
T (chemical analysis)	6.6	5.6

Traces of Ti, Na

% Ash Accounted for (Total Stable Oxides) 95.88

removed and further muffled. Methyl alcohol proved to be a very suitable solvent and cleaner for removing the tars and smoke deposited on the trays. Dilute nitric acid was used to finish the cleaning job. All the residues were ignited for one hour at 1000° C. The residual ash was given a leach with concentrated nitric acid for eight hours and filtered off. The filtrate was given a diethyl ether extraction six times after suitable adjustment of the pH and saturation with $Cu(NO_3)_2$. The combined ether extract was reserved for combination with other layers obtained further on in the process. The acid insolubles were given a fusion treatment in the following manner. The insoluble from the HNO_3 leach was ignited and then fused with $K_2S_2O_7$. The melt was broken up with a treatment with aqua regia, and the leach was filtered off, leaving a small quantity of residue. The leach was re-precipitated three times with NH_4OH and dissolved each time with HNO_3. The final gunk solution was saturated with $Cu(NO_3)_2$ and ether extracted six times. This ether extract and that reserved from the first ether extraction were combined and evaporated over water. The Tuballoy was precipitated from the water layer with NH_4OH and the precipitate was ignited to T_3O_8 and weighed. In the first run the 600 ml. of extracted residue had a concentration of 70 mcg. of T/ml. while in the second run the T concentration in the 500 ml. residue was 4 mcg/ml according to the analysis given by Mr. R. H. Smellie's laboratory.

Application to Plant Scale

The type of recovery described above was fairly simple and could be employed without too much difficulty on a large scale. Every operation except that of fusion is already in use in the area and the fusion is not an impossible step by any means. The gunk, of course, could be extracted by carbitol, as is being done regularly instead of using the diethyl ether. The burning problem has already been discussed incorporating the idea of using an incinerator for preliminary ashing. The filters can, of course, be burned in a muffle as is done in the case of papers at the present time, but it would be a more indirect method of carrying out the operation.

The nitric acid insolubles can be treated in several ways for the effective solution of Tuballoy. Pyrosulfate fusion was used in the experimental reclamation of Tuballoy and proved to be satisfactory. There are two difficulties that detract from its practical application: (1) The sulfate ion must be removed by re-precipitations with ammonia and subsequent solutioning in HNO_3. (2) The silica is not dissolved, but the Tuballoy is put in a soluble form. On a large scale, the fusion melt might not penetrate the larger agglomerates, thus resulting in the incomplete fusion of Tuballoy.

A more satisfactory fusion mixture is found in $K_2CO_3 + Na_2CO_3 + NF$ which is regularly used in Mr. R. H. Smellie's analytical laboratory for solutioning residues with silica content. This mixture is fairly low in melting point and a complete fusion is attained. The fusion mixture after being dissolved in water, acidified and heated, offers no anions that will interfere with ether extraction. The carbonates are removed on boiling. The K_2SiF_6 that is formed in fusion becomes H_2SiF_6 on acidification, and this is easily boiled

off. Any remaining fluoride ion can be effectively complexed with Al^{+++} and extraction carried out with no difficulty. The ash can be put in the muffle at 400° C. with the fusion mixture and the fusion carried out smoothly. This is done in the analytical laboratories at the present time and should prove to be a very effective and worthwhile plant scale method for processing silica-containing residues.

A mixture of Na_2CO_3 and NaOH has been proposed for a fusion mix and might also prove valuable in processing silica residues.

The treatment of the acid insolubles with HF might also be used. In this method the silica would be removed as H_2SiF_6 leaving a soluble residue. The difficulty here would be in getting a resistant container for carrying out the fuming operation. Platinum and tantalum are not attacked, but cheaper metal containers would be to some extent.

It is apparent, however, that T can be suitably removed from ash residues without insuperable difficulty.

RESULTS OF EXPERIMENTS AND APPLICATIONS

Collection Results and Efficiencies

The quantity of Tuballoy found in the first pilot run was 1.975 gms. This includes the 42 milligrams left in the ether extracted residues. From preliminary calculations, 3.1 gms. of Tuballoy was expected. Thus we were able to collect 63.7% of that estimated.

In the second dust collection run the yield was 1.8408 gms. T_3O_8 or 1.56 gms. Tuballoy, while the estimated value was 1.89 gms. Tuballoy. This gives an actual recovery of 82.6% of the quantity estimated. It is interesting also to note that 79% of the Tuballoy recovered was obtained from the acid leach, and the remaining 21% was obtained from the fusion treatment. The actual yield was closer to the calculated theoretical yield in the second run because more data on existing conditions was collected throughout the run than was obtained during the first run.

With the above data it is now possible to say definitely that our method of determining Tuballoy dust concentrations is quite satisfactory. We can with assurance calculate the quantities of Tuballoy present in the air. Also, the quantities of Tuballoy that can be picked up on commercial paper air filters can be easily and accurately predetermined. Dust concentrations that are important from the hygiene aspects can be quickly and accurately determined. These large scale pilot runs have proved that the Tuballoy found in air-borne dust is of an important magnitude and can be readily collected by commercially available methods. The recovery of Tuballoy from process filters also has been shown to be simple and practical.

Calculations for Building 9206

It will be of interest to calculate the amounts of Tuballoy that can be picked up by commercial dust collectors in Building 9206. The concentrations listed in Table V are those found during the months indicated, and may be quite different at the present time, due to the change in procedure and operating conditions. Nevertheless, values can be given which will indicate the order of magnitude of the losses. An overall efficiency factor of 65% will be applied to the calculations as this would seem feasible from the experiences gained in dust collection. Only those rooms showing relatively high Tuballoy concentrations are listed.

The 55.5 gms. of Tuballoy that can be collected on a ten-ply oil-treated filter per day, when figured on a yearly basis, show a total of 20.6 kg. loss. It would seem that this quantity of Tuballoy would be quite worthwhile collecting by the simple manner described in this report.

PARTICLE SIZES

At the time of writing of this report, no particle size measurement data on dusts in Bldg. 9206 is available, but it is the intention of the industrial hygiene group to do some work in this direction. A better knowledge of methods for practical dust collection and a better insight into the dust problem would be gained with the knowledge of size distribution of particles It would be of value especially to know the Tuballoy concentration in the size distribution. This problem is one requiring extensive research and requiring facilities not available to us at the present time.

At this time, however, certain conclusions can be drawn concerning particle size that fairly well answer practical particle size questions. First, the Tuballoy is found in the small as well as the larger particles. We know this because Tuballoy concentration measurements were made at the outlet of the dust collector. The larger particles were presumably caught on the filter, thus allowing only the smaller particles to pass. The average concentration at the filter exhaust was 2.7 mcg/m^3, while the average concentration at the filter inlet was 21 mcg/m^3.

Another consideration is that particle weight varies as the cube of the particle diameter, so that, of course, the bulk of the weight in most industrial dusts is found in the larger particles. Thus, a particle ten times the size of a smaller one will weigh 1,000 times as much as the smaller.

A point that requires considerable attention in the matter of filtration is that the straining out of particles plays a minor part in air filtration. Actually in all filters the open passageways are much larger than the particles that are caught. Furthermore, it has been shown that the power necessary to force air through a filter with pores small enough to strain out particles is very much higher than ever occurs in practice.

According to the authorities on industrial dust, the measurement of the smaller particles that would be involved in this study would be very difficult and of questionable value. The optical difficulties involved in the size measurement of particles less than 1 micron in diameter make particle size measurements of particles less than 1 micron in diameter of questionable value.

For these reasons (along with the fact of the actual experimental collections made) it is felt that the smaller particles require only secondary consideration. If it should be decided that collection of 100% of the dust particles should be attempted, a problem of tremendous magnitude would be presented. It is seen that the bulk of the Tuballoy can be collected economically and easily on a filter. The small residual concentrations of Tuballoy left in the air might be suitably collected on the commercially available electrostatic filter unit which is to be tried out here soon. It should be kept in mind that every

room in the building has some Tuballoy concentration and that processing of all the building air would require a tremendous installation of equipment, requiring many costly ventilation and structural alterations. We have attempted to show through practical pilot runs that the recovery of the bulk of air-borne Tuballoy is relatively simple and quite efficient with the use of filters. We are not able at this time to make recommendations based on practical experiments for the collection of the smaller particles containing a minor portion of air-borne Tuballoy. The lengths to which we should go for the collection of the smaller particles should be suggested by management after consideration of the various factors presented above.

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TABLE V

DUST LOSSES IN 9206

Room No.	T Conc. mcg/m^3	Sampling Period	Volume of Air Exhausted	Expected Collection of T in Gms/day (paper filter)
6	29	(Sept. & Oct.) 1945	8,700	6.76
7	66	" "	2,100	3.69
8	8	(June & July)	16,000	3.42
24	7	" "	7,000	1.31
26	12	" "	3,300	1.06
28	6	" "	6,800	1.08
29	7	" "	3,760	.70
40	27	" "	3,200	2.30
41	273	" "	1,000	7.3
42	32	" "	1,000	.95
43	43	" "	7,200	8.30
44	37	" "	10,350	10.2
45	44	" "	7,200	8.5

55.57 Gms/day

20.6 kg/year

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11-45